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MITSUBISHI GAS CHEM CO INC *JP 2003026638-A
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Manufacture of high purity xylylene diamine used as raw material for polyamide resin, involves adding water and specific solvent, to rough xylylene diamine, and recovering high purity xylylene diamine

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A(1-E5, 8-D3) E(10-B1E, 11-Q1) N(2-A, 2-B, 2-C, 3-C1, 3-D2)

NOVELTY

Xylene is ammoxidated to obtain phthalonitrile. The ammoxidated gas is contacted with organic solvent. Liquid ammonia is added to organic solvent, and hydrogenation is performed. Organic solvent and ammonia are separated from hydrogenation reaction product. Water and aromatic hydrocarbon and/or saturated hydrocarbon solvent, are added to resulting rough xylylene diamine (XD), and high purity XD is recovered.

DETAILED DESCRIPTION

Ammoxidation of raw material xylene is performed by gaseous-phase contact reaction with ammonia and oxygen-containing gas, to obtain a phthalonitrile. The resulting ammoxidation reactive gas is contacted with an organic solvent, directly to collect the phthalonitrile in the organic solvent. Subsequently, liquid ammonia is added to the organic solvent, without separating the phthalonitrile, and hydrogenation reaction is performed. Subsequently, organic solvent and ammonia are separated from the hydrogenation reaction product, to obtain rough xylylene diamine. Water and at least one type of solvent chosen from aromatic hydrocarbon and saturated hydrocarbon, are added to rough xylylene diamine, to separate solvent phase and water phase. Subsequently, high purity xylylene diamine is recovered from the water phase to which extraction-separation is performed. Phthalonitrile is synthesized by ammoxidation reaction of metaxylene or paraxylene.

USE

For manufacture of high purity xylylene diamine used as raw material for polyamide resin and epoxy hardener, and as intermediate raw material of isocyanate.

ADVANTAGE

Xylylene diamine of high purity is obtained efficiently with sufficient yield.

DESCRIPTION OF DRAWING

The figure shows the flowchart of manufacturing method of high purity xylylene diamine. (Drawing includes non-English language text).

EXAMPLE

Ammoxidation of metaxylene was performed by gaseous-phase contact reaction with ammonia and oxygen-containing gas, and phthalonitrile was obtained. The resulting ammoxidation reactive gas was contacted with pseudo cumene organic solvent, and the phthalonitrile was collected in the organic solvent. Subsequently, liquid ammonia was added to the organic solvent, without separating the phthalonitrile, and hydrogenation reaction was performed. Subsequently, organic solvent and ammonia were separated from the hydrogenation reaction product, and rough metaxylylene diamine was collected. Water was added to the rough metaxylylene diamine, to separate solvent phase and water phase. Subsequently, high purity metaxylylene diamine was recovered from the water phase. Batch distillation of metaxylylene diamine containing water phase was performed, and water was separated, and high purity xylylene diamine with purity of 99.99 wt.%, was obtained.

TECHNOLOGY FOCUS

Organic Chemistry - Preferred Solvent: The organic solvent is an aromatic hydrocarbon. Preferred Catalyst: Manufacture of high purity xylylene diamine is performed using a fluid catalyst containing one or more types of metallic oxide chosen from vanadium, molybdenum and iron. Hydrogenation is performed in presence of nickel and/or cobalt catalyst.

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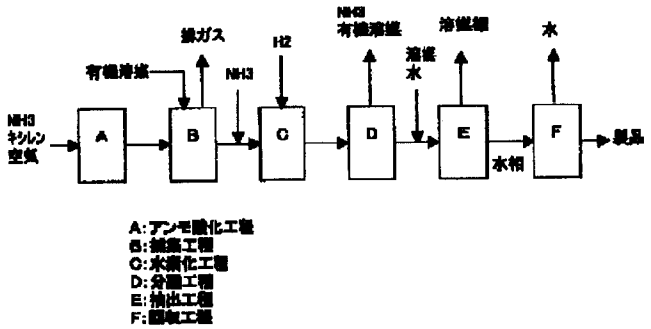

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